

الآية

بسم الله الرحمن الرحيم

: قال تعالى

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صدق الله العظيم

سورة العلق

(الآية 1-5)

I dedicate this work to

My parents

My family

I would like to express my sincere appreciation and gratitude to my supervisor Dr. Ahmed Elsadig Mohamed Saeed for his lucid design of this research and help throughout his work. I wish to thank U. Fathi Abbas for being available willingly to provide help when being asked. My thanks also to U. Amira Abd Elaziz for taking my samples to Cairo for H^1 -NMR and M.S analysis. Finally I am grateful to the Chemistry laboratory staff for their great help.

Abstract

Thiadiazole compounds are playing an important role in our life, through their biological activities in various fields, as antibacterial, antifungal, plant activator, and as pesticides. In this work six compounds as 1,2,3-thiadiazole derivatives were prepared. In this work carbonyl groups were allowed to condense with ammonia derivatives of semicarbazide to give imines' bond. The carbonyl group of semicarbazide acts as a reactive group in cyclization of the reaction product between ketonic compounds of semicarbazide (semicarbazone) with thionyl chloride to give five-membered heterocyclic rings. The compounds were prepared according to a well-established literature methods and the identities of these compounds were confirmed by IR, H^1 -NMR, and MS Spectrometer and T.L.C. analysis. Mechanism of reaction and appropriate retro synthetic analysis were given and discussed in chapter three. Detailed spectral (IR, H^1 -NMR, MS) analysis of the target compounds was given. The functional groups and protons environment were assigned to the proper absorption bonds, special emphasis was put upon the carbonyl group stretching vibration in IR. A variation in the number of peaks, positions and increasing or decreasing in the values of bonds were noticed according to the various effects, such as resonance effects, conjugation effects and anisotropic effects.

بسم الله الرحمن الرحيم

خلاصة البحث

مركبات الثنائي آزول تمثل مركبات حلقة غير متجانسة هامة جداً في الحياة. تتمثل أهميتها في أنها تدخل في الأنشطة الحيوية. كمضاد للبكتيريا، الفطريات، تعمل علي تقليل السموم، تساعد علي نمو النباتات، وأيضاً تستخدم كمبيدات. في هذا العمل تم تحضير ست مركبات عضوية عبارة عن مشتقات 1,2,3- ثنائي ثيا آزول كناتج أخير. في هذا العمل تم تكاثف مجموعة الكربونيل الكيتونية (الليفاتية منها والاروماتية) مع مشتق الأمونيا محتوية أيضاً علي مجموعة كربونيلية (سمي كاربازيد) لإعطاء مركب كوسيط في مرحلة التحضير ذات رابطة تسمي (إمين). أهمية الزمرة الكربونيلية في مركب السمي كاربازيد تتمثل في أنها تمثل كمجموعة مغادرة جيدة في تفاعل تكوين الحلقة الغير متجانسة كناتج أخير في موضوع البحث، والذي نتج من تفاعل التكاثف (سمي كاربازون) مع مركب الثيونايل كلورايد لإعطاء حلقة خماسية غير متجانسة (1,2,3- ثنائي ثيا آزول). هذا المركب الناتج تم تحضيره وفقاً لأسس نظرية وطرق تحضيرية مسبقة وسليمة، وقد تم تحديد نسبته و هويتها وبنيتها التركيبية عن طريق الطرق الطيفية المتمثلة في الأشعة تحت الحمراء لمعرفة الزمر الوظيفية في الحلقة وطيف الرنين النووي المغناطيسي لمعرفة بيئات الهيدروجين فيها وأيضاً طيف الكتلة لمعرفة الوزن الجزيئي وكروماتوغرافيا الطبقة الرقيقة لمعرفة تمام تكوين الحلقة كمركب جديد. ميكانيكية التفاعل والتخليق الرجعي لهذه المركبات نوقشت في الباب الثالث بالإضافة الي تفاصيل التحليل الطيفي لكل من طيف الأشعة تحت الحمراء وطيف الرنين النووي المغناطيسي و طيف الكتلة والتي تتمثل في المجموعات الوظيفية وبيئات الهيدروجين في شكل حزم إمتصاص خاصة حزم إمتصاص مجموعة الكربونيل في طيف الأشعة تحت الحمراء الإمتطاطية منها. الإختلاف في عدد الحزم وأماكنها والزيادة والنقصان في قيم حزم الإمتصاص لوحظت وفقاً للإختلاف في العوامل المؤثرة علي حزم الإمتصاص في الشكل والقيمة لهذه الحزم كل من الأثر الرنيني، الأثر التتابعي، الأثر الأنيزوتروبي والروابط الأليية الموجودة في المركب.

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List of abbreviations

abbreviation	Meaning of the abbreviation	Page no.
COX-2	Cyclooxygenase	24
Comp. number	Compound number.	34
M. formula	Molecular formula	34
M. wt.	Molecular weight.	34
Acetanill.	Acetanilide.	37
acetoph.	acetophenone.	37
acetoph. Semicarb.	acetophenone semicarbazone	37
chloro.sulph.	chlorosulphonic acid	37
DEE	Diethyl ether.	37
M.P.	Melting point.	37
p-amino acetoph.	p- amino acetophenone	37
p- amino acetoph. semicarb.	p- amino acetophenone semicarbazone.	38
4- (P-acetamido acetoph. semicarb.)	4- (p- acetamido acetophenone semicarbazone.	38
p- amino acetoph.	p- amino acetophenone.	38
p- acetamido acetoph.	p- acetamido acetophenone.	38
p- benzamido acetoph.	p- benzamido acetophenone	38
4-(p-acetamidoph.sulph.acetoph.)	4-(p-acetamido phenyl sulphonamido acetophenone.	38
perc. yield	percentage yield	38
Reac. temp.	Reaction temperature	38
Rec. solv.	Recrystallization solvent	38
Semicarb.	semicarbazide	38
thion. Chloride	thionyl chloride	39
Und.ref.	under reflux	38
Bend.	Bending.	42

St.vib.	Stretching vibration.	42
Abs. ethanol	absolute ethanol	40
RSA	retro synthetic analysis	48